

A

 1530 U.S. PRO  
 09/34334  
 06/30/99

# UTILITY PATENT APPLICATION TRANSMITTAL

(Only for new nonprovisional applications under 37 CFR 1.53(b))

Box PATENT APPLICATION  
Assistant Commissioner for Patents  
Washington, DC 20231

Case Docket No. T-5586CIP  
Chevron Corporation  
Law Department - Patent and Licensing Unit  
P. O. Box 7141  
San Francisco, CA 94120-7141

"Express Mail" mailing label No. EE660535429US  
Date of Deposit June 30, 1999

I hereby certify that this paper or fee is being deposited with the United States Postal Service "Express Mail Post Office to Addressee" service under 37 CFR 1.110 on the date indicated above and is addressed to Box PATENT APPLICATION, Assistant Commissioner for Patents, Washington, DC 20231.

Jadine Jay  
(Typed or printed name of person)

(Signature of person mailing paper or fee)

Sir:

Transmitted herewith for filing is the patent application of:

Inventor: GREGG M. SKLEDAR ET AL.

For: POLYALPHAOLEFINS WITH  
IMPROVED OXIDATIVE STABILITY AND  
THE PROCESS OF MAKING THEREOF

Enclosed are:

- ☒ Specification [Total Pages = 22].
- ☐ An Assignment of the invention to **Chevron Chemical Company LLC**, San Francisco, CA, a corporation of Delaware.
- ☐ A certified copy of a \_\_\_\_\_ application.
- ☒ Drawing(s) [Total Sheets = 1].
- ☐ Combined Oath or Declaration and Power of Attorney [Total Pages = \_\_\_\_].
- ☐ Newly executed (original or copy)
- ☐ Copy from a prior application (37 CFR 1.63(d)) (for continuation/divisional)
- ☐ **DELETION OF INVENTOR(S)**  
Signed statement attached deleting inventor(s) named in the prior application, see 37 CFR 1.63(d)(2) and 1.33(b).
- ☐ Incorporation by Reference (*useable if copy of Oath or Declaration from prior application is being submitted*)  
The entire disclosure of the prior application, from which a copy of the oath or declaration is being supplied, is considered as being part of the disclosure of the accompanying application and is hereby incorporated by reference therein.
- ☐ Information Disclosure Statement.
- ☒ If a CONTINUING APPLICATION, check appropriate box and supply the requisite information:
  - ☐ Continuation ☐ Divisional ☒ Continuation-in-part (CIP) of prior application No: 09/108,048

The filing fee has been calculated as shown below:

	(Col. 1)	(Col. 2)
For	No. Filed	No. Extra
Basic Application Fee		
Total claims	28 - 20	8
Independent claims	3 - 3	0
<input type="checkbox"/> Multiple dependent claims presented		

\*If the difference in Col. 1 is less than zero, enter "0" in Col. 2

Rate	Fee
	\$760.00
x \$18.00 =	\$144.00
x \$78.00 =	\$ 0.00
+ \$260.00 =	\$ 0.00
TOTAL	\$904.00

- ☒ Please charge Deposit Account No. 03-1620 in the amount of **\$904.00**. A triplicate copy of this sheet is enclosed.
- ☒ The Assistant Commissioner is hereby authorized to charge payment of the following fees associated with this communication or during the pendency of this application or credit any overpayment to Deposit Account No. 03-1620. A triplicate copy of this sheet is enclosed.
  - ☒ Any additional filing fees required under 37 CFR 1.16.
  - ☒ Any patent application processing fees under 37 CFR 1.17.
  - ☒ Any filing fees under 37 CFR 1.16 for presentation of extra claims.
  - ☒ Any extension of time fees under 37 CFR 1.36(a).

WBHaymond:jj  
Enclosures

June 30, 1999

116930

W. Bradley Haymond  
Reg. No. 35,186  
Attorney of Record  
P.O. Box 7141, San Francisco, CA 94120-7141  
Area Code (415) 894-2471

1 BE IT KNOWN, that We, GREGG M. SKLEDAR, a citizen of the United  
2 States of America, resident of Kingwood, County of Harris, State of Texas,  
3 and KENNETH D. HOPE, a citizen of the United States of America, resident  
4 of Kingwood, County of Harris, State of Texas, have invented new and useful  
5 improvements in

6 **POLYALPHAOLEFINS WITH IMPROVED OXIDATIVE STABILITY**  
7 **AND THE PROCESS OF MAKING THEREOF**

8

9 **CROSS-REFERENCE TO RELATED APPLICATIONS**

10 This application is a Continuation-In-Part of U.S. Application Serial  
11 No. 09/108,048, filed June 30, 1998.

**POLYALPHAOLEFINS WITH IMPROVED OXIDATIVE STABILITY  
AND THE PROCESS OF MAKING THEREOF**

**CROSS-REFERENCE TO RELATED APPLICATIONS**

This application is a Continuation-In-Part of U.S. Application Serial  
No. 09/108,048, filed June 30, 1998.

**FIELD OF THE INVENTION**

The present invention relates to oxidatively stable polyalphaolefins and  
lubricating composition comprising same. More particularly, the present  
invention relates to compositions of lubricants using synthetic  
polyalphaolefins derived from 1-decene, 1-dodecene or 1-tetradecene olefins  
which exhibit improved oxidative stability.

**BACKGROUND OF THE INVENTION**

Lubricants today are being called upon to work in ever more demanding  
applications. In many applications, greater thermal and oxidative  
performance are necessary to meet rigorous requirements. For instance,  
today's automobiles tend to have smaller, more demanding engines that  
operate at higher temperatures. Thus, the engine oil has to function in an  
increasingly severe environment while meeting fuel economy demands.  
Besides changes in the additive package, increasingly synthetic base oils are  
being used instead of conventional mineral oils. Of the synthetic oils,  
polyalphaolefins (PAO) are among the most popular.

PAO is manufactured by oligomerization of linear alpha olefin followed by  
hydrogenation to remove unsaturated moieties and fractionation to obtain the  
desired product slate. 1-decene is the most commonly used alpha olefin in

1 the manufacture of PAO, but 1-dodecene and 1-tetradecene can also be  
2 used. PAO's are commonly categorized by the numbers denoting the  
3 approximate viscosity in centistokes of the PAO at 100°C. It is known that  
4 PAO 2, PAO 2.5, PAO 4, PAO 5, PAO 6, PAO 7, PAO 8, PAO 9 and PAO 10  
5 and combinations thereof can be used in engine oils, gear oils, compressor  
6 lubricants, hydraulic fluids and a variety of other applications. The most  
7 common of these are PAO 4, PAO 6 and PAO 8.

8 It has long been known that hydrogenation to achieve a PAO which is  
9 predominantly saturated achieves a more desirable product, one that is more  
10 stable to oxidation and heat.

11 Several patents disclose processes for hydrogenating PAO's. These include  
12 the following:

13 Jackson et al. (U.S. Patent No. 4,125,569) discloses a process for  
14 hydrogenating polymerized olefins in the presence of alumina and a  
15 hydrogenated catalyst to provide a greater hydrogenation rate than what is  
16 obtained using the catalyst alone.

17 Petrillo et al. (U.S. Patent No. 4,167,534) discloses the possibility of generally  
18 improving stability to both oxidation and heat as well as specifically improving  
19 viscosity index and pour point by including a hydrogenation step to eliminate  
20 unsaturations in the process of synthesizing lubricating oils from an n-olefin  
21 cut. It does not give any data supporting its general assumption that  
22 oxidative stability of the lubricant is improved by decreasing unsaturation.

23 Degnan et al. (U.S. Patent No. 5,573,657) discloses hydrogenating lubricants  
24 using a catalyst based on an ultra-large-pore crystalline material.

1 With today's automobiles, engine oils and lubricants with high oxidative  
2 stability are needed. Various tests to measure oxidative stability are known.  
3 These include the Lube Oil Oxidator test, the Rotary Bomb Oxidation Test  
4 (RBOT), and the Penn State Microoxidation Test among others. Using such  
5 tests, attempts have been made to correlate increased oxidative stability with  
6 other components or factors in the oil or lubricant.

7 Ripple and Fuhrmann in "Performance Comparisons of Synthetic and Mineral  
8 Oil Crankcase Lubricant Base Stocks" (*Journal of Synthetic Lubricants*, 6-3,  
9 pp. 209-232, 1989) state that engine oil formulations using synthetic (PAO)  
10 base oils provide for superior performance to mineral oils in bench and engine  
11 testing as well as field service testing. This is due to the fluid's increased  
12 oxidative stability, reduced oil consumption, cleaner engines and longer drain  
13 capabilities. Specifically, the oxidative stability is supported by reduced  
14 viscosity increase. But there is nothing in the paper, which attributes this  
15 increase to any specific physical property such as decreased unsaturation as  
16 measured by Bromine Index.

17 Gonsel et al. in "Evaluation of Some Poly-Alpha-Olefins in a Pressurized  
18 Penn State Microoxidation Test" (*Journal of the Society of Tribologists and  
19 Lubrication Engineers*, 43, 8, pp. 629-635, 1987) compared two PAO's, one  
20 with a stated Bromine Index of 1323 and the other with a stated Bromine  
21 Index of two. With a Penn State Microoxidation test with two additive  
22 packages, phenyl alpha naphthylamine (PAN) and zinc dialkyl dithio  
23 phosphate (ZDDP), side by side results of 1% PAN and 1.88% ZDDP/0.5%  
24 PAN show that there may be some slight improvement in oxidative stability for  
25 PAO having extremely low Bromine Index over PAO having a relatively high  
26 Bromine Index, but the advantage does not appear significant since there is  
27 so much scatter in the data that there ends up being no difference  
28 statistically.

1 Even though teachings in the art generally support the presumption that a  
2 decrease in unsaturation in the PAO contained in PAO based lubricants will  
3 have some improving effect on oxidation stability, when such an effect was  
4 actually tested by Gonsel et al., the effect was found to be slight. Therefore,  
5 the art is devoid of the significant benefit realized by greatly hydrogenating  
6 PAO's to increase oxygen stability in PAO based lubricants.

## 7 SUMMARY OF THE INVENTION

8 The present invention relates to a highly oxidative stable polyalphaolefin  
9 method of producing a highly oxidatively stable polyalphaolefin comprising the  
10 step of hydrogenating a polyalphaolefin to a level of hydrogenation in which a  
11 Bromine Index of less than 200 mg Bromine per 100 gram sample of  
12 polyalphaolefin is achieved. In more preferred embodiments, the present  
13 invention relates to the above method in which a Bromine Index of less than  
14 100 mg Bromine per 100 gram sample of polyalphaolefin is achieved, a  
15 Bromine Index of less than 50 mg Bromine per 100 gram sample of  
16 polyalphaolefin is achieved, and a Bromine Index of less than 25 mg Bromine  
17 per 100 gram sample of polyalphaolefin is achieved.

18 In another embodiment, the present invention relates to a lubricating  
19 composition comprised of the highly oxidative stable polyalphaolefin. The  
20 present invention, and the benefits realized in its practice, is based at least in  
21 part on the recognition that by going to near complete hydrogenation, one  
22 achieves a surprising improvement in oxidative stability for polyalphaolefins.

## 23 DESCRIPTION OF THE FIGURE

24 The Figure of the Drawing plots the results of experiments showing oxidation  
25 over time in a Penn State Microoxidation Test of two PAO's (a typical  
26 moderately hydrogenated PAO and a highly saturated product) along with

1 results given in Gonsel et al. The GPC data, which has been analyzed by the  
2 inventors, has been modified to fit the scale of Figure 2 in Gonsel et al.

3 DETAILED DESCRIPTION OF THE INVENTION

4 Investigators continue to search for ways to increase oxidation stability in  
5 PAO's. The inventors in the present application have found a surprising  
6 increase in oxidative stability as a result of hydrogenating PAO's to decrease  
7 unsaturation to a Bromine Index below 200. The Bromine Index (ASTM  
8 D 2710) is the number of milligrams of Bromine that react with 100 grams of  
9 sample under the conditions of the test. In contrast, the Bromine Number  
10 method (ASTM D 1159), as mentioned in Petrillo et al. (U.S. Patent  
11 No. 4,167,534) is the number of grams that react with 100 grams of sample  
12 under the conditions of the test. Therefore, there is a natural factor of 1000  
13 difference between the two methods. This increase in oxidative stability is  
14 measured with both Rotary Bomb Oxidation Test (RBOT) (ASTM D 2272) and  
15 Lube Oil Oxidator tests. The degree of increase of oxidative stability  
16 conferred by the hydrogenation step is far beyond what would be expected  
17 from previous teachings in the art.

18 The Bromine Index method, ASTM D 2710, was developed to determine the  
19 degree of unsaturation in petroleum hydrocarbons, such as cumenes,  
20 reformates and kerosenes. Nevertheless, it has historically been utilized as a  
21 measure for the degree of unsaturation for PAO's. The chemical structure of  
22 PAO differs from the aforementioned petroleum hydrocarbons in terms of the  
23 degree of branching and therefore there is a greater steric hindrance to the  
24 bromination reaction for PAO. In addition, PAO is limited in solubility in the  
25 test solvent, which creates problems with accuracy and repeatability.  
26 Therefore, the Bromine Index method has been modified from the original  
27 ASTM D 2710 specifically for PAO and is designated as K801.

1 The present inventors have found specific modifications useful in increasing  
2 the accuracy and repeatability and decreasing the level of detection for PAO  
3 measurements. The modifications have been to utilize isopropanol as an  
4 additional solvent and operate at higher temperatures to aid solubility as well  
5 as take blank measurements for each titration solvent. In addition, the  
6 present inventors have also identified that it is preferred to utilize a specific  
7 instrument vendor (Mettler) for the Bromine Index apparatus. This has  
8 resulted in improvements in both accuracy and repeatability based upon  
9 measurement standards for PAO.

10 The present inventors used the above modifications to the original Bromine  
11 Index method to more accurately determine Bromine Index of a moderately  
12 hydrogenated PAO product and a highly saturated product and then  
13 determined oxidative stability of these products using the Penn State  
14 Microoxidation method under the same equipment and under the same  
15 conditions as in Gunsel et al.'s Figure 2, as described in Example 11  
16 hereafter. The present inventors found that the oxidative stability of both a  
17 moderately hydrogenated PAO product (having a Bromine Index of 433  
18 measured with the modified method) and a highly saturated PAO product  
19 (with a Bromine Index of 0.95 measured with the modified method) was  
20 significantly better than the oxidative stability of Gunsel et al.'s Fluid B (a PAO  
21 fluid having a stated Bromine Index of 2 measured by the unmodified Bromine  
22 Index method). Thus, Gunsel et al.'s hydrogenated PAO (Fluid 2) was not  
23 nearly as oxidatively stable as either a typical hydrogenated PAO product with  
24 a Bromine Index of 433 or a highly saturated PAO product with a Bromine  
25 Index of 0.95 in accordance with the present invention, as measured by the  
26 improved Bromine Index measurement methods.

27 The present inventors have also found that when they hydrogenate the PAO  
28 twice, both before and after a distillation step, they achieve a better result in



1 both the RBOT and Lube Oil Oxidator tests than they do when only one  
2 hydrogenation step is performed with a distillation step.

3 In its broadest aspect, the present invention involves improving thermal  
4 oxidative stability by hydrogenating PAO's especially those derived from  
5 1-decene, 1-dodecene or 1-tetradecene as a base oil. The level of  
6 hydrogenation preferably approaches the removal of all unsaturation, but is at  
7 least to a level such that the Bromine Index is less than 200 mg Bromine per  
8 100 gram of polyalphaolefin.

9 The PAO's described in the present invention can be used, as in the following  
10 non-limiting examples, as engine oil lubricant, gear lubricant, hydraulic  
11 lubricant, compressor lubricant, aerospace jet lubricant, fiber optic cable gel,  
12 synthetic grease, and dielectric fluid.

13 The present invention also relates to a method of producing a highly  
14 oxidatively stable polyalphaolefin comprising the step of hydrogenating  
15 polyalphaolefin to a level of hydrogenation in which an RBOT level of at least  
16 2200 minutes is achieved when diphenyl amine is used as an antioxidant.  
17 This is illustrated in Examples 1-8.

18 The present invention also relates to a method of producing a highly  
19 oxidatively stable polyalphaolefin comprising the step of hydrogenating  
20 polyalphaolefin to a level of hydrogenation in which a Lube Oil Oxidator level  
21 of at least 45 hours is achieved when pressures between 100 and 2500 psi  
22 are applied. This is illustrated in Examples 9 and 10.

23 The present invention also relates to a method comprising distilling the  
24 polyalphaolefin to remove impurities, then hydrogenating the polyalphaolefin  
25 to achieve a final polyalphaolefin product having a Bromine Index of less than

1 200 mg Bromine per 100 gram sample of polyalphaolefin. This is illustrated in  
2 Example 9.

3 The present invention also relates to a method comprising a preliminary step  
4 of hydrogenating the polyalphaolefin followed by distilling to remove  
5 impurities, followed by a hydrogenating step to achieve a final polyalphaolefin  
6 product having a Bromine Index of less than 200 mg Bromine per 100 gram  
7 sample of polyalphaolefin. This is illustrated in Example 10, and is a  
8 preferred embodiment of the present invention.

### 9 ADDITIVE COMPONENTS

10 The following additive components are examples of some components that  
11 can be favorably employed in the preparation of the lubricating composition in  
12 accordance with the present invention. These examples of additives are  
13 provided to illustrate the present invention, but they are not intended to limit it:

14 (1) Metal detergents: sulfurized or unsulfurized alkyl or alkenyl phenates,  
15 alkyl or alkenyl aromatic sulfonates, sulfurized or unsulfurized metal  
16 salts of multi-hydroxy alkyl or alkenyl aromatic compounds, alkyl or  
17 alkenyl hydroxy aromatic sulfonates, sulfurized or unsulfurized alkyl or  
18 alkenyl naphthenates, metal salts of alkanolic acids, metal salts of an  
19 alkyl or alkenyl multi-acid, metal salts of an alkyl salicylic acid,  
20 carboxylates, overbased detergents and chemical and physical mixtures  
21 thereof.

22 (2) Ashless dispersants: alkenyl succinimides, alkenyl succinimides  
23 modified with other organic compounds, and alkenyl succinimides  
24 modified with boric acid, alkenyl succinic ester.

1 (3) Oxidation inhibitors:

- 2 (a) Phenol type oxidation inhibitors: 4,4'-methylenebis (2,6-di-tert-  
3 butylphenol), 4,4'-bis(2,6-di-tert-butylphenol), 4,4'-bis(2-methyl-6-  
4 tert-butylphenol), 2,2'-(methylene bis (4-methyl-6-tert-butyl-phenol),  
5 4,4'-butylidenebis(3-methyl-6-tert-butylphenol),  
6 4,4'-isopropylidenebis(2,6-di-tert-butylphenol), 2,2'-methylenebis(4-  
7 methyl-6-nonylphenol), 2,2'-isobutylidene-bis(4,6-dimethylphenol),  
8 2,2'-methylenebis(4-methyl-6-cyclohexylphenol), 2,6-di-tert-butyl-  
9 4-methyl-phenol, 2,6-di-tert-butyl-4-ethylphenol, 2,4-dimethyl-6-  
10 tert-butyl-phenol, 2,6-di-tert-4-(N,N' dimethylaminomethylphenol),  
11 4,4'-thiobis(2-methyl-6-tert-butylphenol), 2,2'-thiobis(4-methyl-6-  
12 tert-butylphenol), bis(3-methyl-4-hydroxy-5-tert-butylbenzyl)-  
13 sulfide, and bis (3,5-di-tert-butyl-4-hydroxybenzyl).
- 14 (b) Diphenylamine type oxidation inhibitor: alkylated diphenylamine,  
15 phenyl-I-naphthylamine, and alkylated I-naphthylamine.
- 16 (c) Other types: metal dithiocarbamate (e.g., zinc dithiocarbamate),  
17 and methylene bis (dibutyl dithio carbamate).

18 (4) Rust inhibitors (Anti-rust agents):

- 19 (a) Nonionic polyoxyethylene surface active agents: polyoxyethylene  
20 lauryl ether, polyoxyethylene higher alcohol ether, polyoxyethylene  
21 nonylphenyl ether, polyoxyethylene octylphenyl ether,  
22 polyoxyethylene octyl stearyl ether, polyoxyethylene oleyl ether,  
23 polyoxyethylene sorbitol monostearate, polyoxyethylene sorbitol  
24 mono-oleate, and polyethylene glycol monooleate.

- 1 (b) Other compounds: stearic acid and other fatty acids, dicarboxylic  
2 acids, metal soaps, fatty acid amine salts, metal salts of heavy  
3 sulfonic acid, partial carboxylic acid ester of polyhydric alcohol, and  
4 phosphoric ester.
- 5 (5) Demulsifiers: addition product of alkylphenol and ethylene oxide,  
6 polyoxyethylene alkyl ether, and polyoxyethylene sorbitan ester.
- 7 (6) Extreme pressure agents (EP agents): zinc dithiophosphates, zinc  
8 dithiocarbamates, zinc dialkyl dithiophosphate (primary alkyl type &  
9 secondary alkyl type), zinc diaryl dithiophosphate, sulfurized oils,  
10 diphenyl sulfide, methyl trichlorostearate, chlorinated naphthalene,  
11 fluoroalkylpolysiloxane, and lead naphthenate.
- 12 (7) Friction modifiers: fatty alcohol, fatty acid, amine, borated ester, and  
13 other esters.
- 14 (8) Multifunctional additives: sulfurized oxymolybdenum dithiocarbamate,  
15 sulfurized oxymolybdenum organo phosphoro dithioate,  
16 oxymolybdenum monoglyceride, oxymolybdenum diethylate amide,  
17 amine-molybdenum complex compound, and sulfur-containing  
18 molybdenum complex compound.
- 19 (9) Viscosity index improvers: polymethacrylate type polymers, ethylene-  
20 propylene copolymers, styrene-isoprene copolymers, hydrated styrene-  
21 isoprene copolymers, polyisobutylene, and dispersant type viscosity  
22 index improvers.
- 23 (10) Pour point depressants: polymethyl methacrylate.

1 (11) Foam Inhibitors: alkyl methacrylate polymers and dimethyl silicone  
2 polymers.

3 In one embodiment, an engine lubricating oil composition would contain:

4 (a) a major part of a base oil of lubricating viscosity, wherein the base oil  
5 comprises 1-dodecene and/or 1-tetradecene-derived polyalphaolefins;

6 (b) 0% to 20% of at least one ashless dispersant;

7 (c) 0% to 30% of the detergent;

8 (d) 0% to 5% of at least one zinc dithiophosphate;

9 (e) 0% to 10% of at least one oxidation inhibitor;

10 (f) 0% to 1% of at least one foam inhibitor; and

11 (g) 0% to 20% of at least one viscosity index improver.

12 In a further embodiment, an engine lubricating oil composition is produced by  
13 blending a mixture of the above components. The lubricating oil composition  
14 produced by that method might have a slightly different composition than the  
15 initial mixture, because the components may interact. The components can  
16 be blended in any order and can be blended as combinations of components.

#### 17 ADDITIVE CONCENTRATES

18 The use of additive concentrates is also included within the scope of this  
19 invention. The concentrates of this invention comprise the compounds or  
20 compound mixtures of the present invention, with at least one of the additives

1 disclosed above. Typically, the concentrates contain sufficient organic diluent  
2 to make them easy to handle during shipping and storage.

3 From 20% to 80% of the concentrate is organic diluent. Suitable organic  
4 diluents which can be used include for example, solvent refined 100N, i.e.,  
5 Cit-Con 100N, and hydrotreated 100N, i.e., RLOP 100N, and the like. The  
6 organic diluent preferably has a viscosity of from about 1 to about 20 cSt at  
7 100°C.

### 8 EXAMPLES

9 The invention will be further illustrated by following examples, which set forth  
10 particularly advantageous method embodiments. While the Examples are  
11 provided to illustrate the present invention, they are not intended to limit it.

12 Examples 1 through 4 are comparative examples, which show typical  
13 oxidative stability results for the described materials. Examples 5 through 8  
14 are intended to show the advantages of the present invention.

#### 15 Example 1

16 A commercial sample of Chevron 4 cSt polyalphaolefin Synfluid® obtained  
17 and subjected to RBOT (ASTM D 2272), the aforementioned modified  
18 Bromine Index and Lube Oil Oxidator measurements. The Lube Oil Oxidator  
19 measurement is an oxygen uptake test wherein the amount of time is  
20 measured until one liter of oxygen is consumed by the sample under the  
21 conditions of the test. Under the conditions of the test, the sample is  
22 formulated with an oxidation catalyst to promote oxidation and an antioxidant  
23 at a controlled temperature and pressure. The RBOT test is an oxygen  
24 uptake test, which monitors pressure changes in a sample bomb at elevated

1 temperature and pressure in the presence of a copper oxidation catalyst. The  
2 results are shown in Table 1.

3 TABLE 1

4

Example	PAO Grade	RBOT*, min	Lube Oil Oxidator, hrs	Bromine Index
1	4	1267	16	682
2	6	826	16	433
3	5	1883	27	172
4	7	1918	25	108
5	4	2214	48	2.6
6	6	1905	>50	1.6
7	5	2233	57	10
8	7	2217	44	5

5  
6 \*All of the samples for RBOT were formulated with 0.5 weight percent of  
7 Uniroyal's Naugalube® 640 antioxidant.

8  
9 Example 2

10 The procedure of Example 1 was repeated except 6 cSt polyalphaolefin was  
11 utilized instead of 4 cSt polyalphaolefin. The results are shown in Table 1.

12 Example 3

13 The procedure of Example 1 was repeated except 5 cSt polyalphaolefin was  
14 utilized instead of 4 cSt polyalphaolefin. The results are shown in Table 1.

15 Example 4

16 The procedure of Example 1 was repeated except 7 cSt polyalphaolefin was  
17 utilized instead of 4 cSt polyalphaolefin. The results are shown in Table 1.

## 1

2

## 9

10

## 12

13

## 16

17

## 19

20



1 Oxidator method. The results are shown in Table 2. The 4 cSt fluid is listed  
2 as Example 9a and the 6 cSt fluid is listed as Example 9b.

3 Example 10

4 The procedure of Example 9 was repeated except an additional  
5 hydrogenation step was carried out prior to the distillation step. The material  
6 was the tested by the Lube Oil Oxidator method. The results are shown in  
7 Table 2. The 4 cSt fluid is listed as Example 10a and the 6 cSt fluid is listed  
8 as Example 10b.

9 TABLE 2

Example	Lube Oil Oxidator, hrs
9a	46
9b	42
10a	30
10b	30

10

11 Example 11

12 Experiments were performed using the Penn State Microoxidation test  
13 methods described in Gunsel et al. to compare oxidation over time of typical  
14 moderately hydrogenated PAO (Bromine Index is 433) and highly saturated  
15 PAO product (Bromine Index is 0.95) with the data described in Gunsel et al  
16 with regard to Fluid B (stated Bromine Index = 2). The results are plotted in  
17 the Figure and show that compared to the results of Fluid B of Gunsel et al.,  
18 there is substantially less formation of high molecular weight product for either  
19 the typical moderately hydrogenated PAO or the highly saturated PAO in a  
20 Penn State Microoxidation test for PAO's containing 1% phenyl alpha  
21 naphthyl amine (PAN). The Microoxidation test was performed in the

1 identical equipment used by Gunsel et al. at Pennsylvania State University.  
2 The test procedure was performed at 225°C and at atmospheric pressure as  
3 described in Figure 2 of Gunsel et al. The GPC analysis was performed  
4 under the same conditions as in Gunsel et al.

5 These results definitively show that the PAO described in Gunsel et al. as  
6 having a Bromine Index of 2 is a material which lacks the oxidative stability of  
7 the PAO of the present invention. In fact, the PAO described in Gunsel et al.  
8 exhibits less oxidative stability than the moderately hydrogenated PAO  
9 (Bromine Index = 433). The effect of saturation of PAO's on oxidative stability  
10 is significantly and unexpectedly greater than what is taught in Gunsel et al.  
11 This also shows that the Bromine Index of 2 in Fluid B reported by Gunsel  
12 et al. is incorrect when specifically tested using modifications of the Bromine  
13 Index method which resolve limitations in the capability of measuring low  
14 Bromine Indices.

15 While the present invention has been described with reference to specific  
16 embodiments, this application is intended to cover those various changes and  
17 substitutions that may be made by those skilled in the art without departing  
18 from the spirit and scope of the appended claims.

1 WHAT IS CLAIMED IS:

- 2 1. A method of producing a high oxidative stability polyalphaolefin  
3 comprising the step of hydrogenating polyalphaolefin to a level of  
4 hydrogenation in which a Bromine Index of less than 200 mg Bromine  
5 per 100 gram sample of polyalphaolefin is achieved.
- 6 2. A method according to Claim 1 wherein a Bromine Index of less than  
7 100 mg Bromine per 100 gram sample of polyalphaolefin is achieved.
- 8 3. A method according to Claim 1 wherein a Bromine Index of less than  
9 50 mg Bromine per 100 gram sample of polyalphaolefin is achieved.
- 10 4. A method according to Claim 1 wherein a Bromine Index of less than  
11 25 mg Bromine per 100 gram sample of polyalphaolefin is achieved.
- 12 5. A method according to Claim 1 further comprising distilling the  
13 polyalphaolefin to remove impurities before the hydrogenating step.
- 14 6. A method according to Claim 5 wherein a Bromine Index of less than  
15 100 mg Bromine per 100 gram sample of polyalphaolefin is achieved.
- 16 7. A method according to Claim 5 wherein a Bromine Index of less than  
17 50 mg Bromine per 100 gram sample of polyalphaolefin is achieved.
- 18 8. A method according to Claim 5 wherein a Bromine Index of less than  
19 25 mg Bromine per 100 gram sample of polyalphaolefin is achieved.
- 20 9. A method according to Claim 5 further comprising a preliminary  
21 hydrogenating of the polyalphaolefin before the distilling step.

- 1 10. A method according to Claim 9 wherein a Bromine Index of less than  
2 100 mg Bromine per 100 gram sample of polyalphaolefin is achieved.
- 3 11. A method according to Claim 9 wherein a Bromine Index of less than  
4 50 mg Bromine per 100 gram sample of polyalphaolefin is achieved.
- 5 12. A method according to Claim 9 wherein a Bromine Index of less than  
6 25 mg Bromine per 100 gram sample of polyalphaolefin is achieved.
- 7 13. A lubricant composition comprising a polyalphaolefin having a Bromine  
8 Index of less than 200 mg Bromine per 100 gram sample of  
9 polyalphaolefin.
- 10 14. A composition according to Claim 13 wherein the composition has a  
11 Bromine Index of less than 100 mg Bromine per 100 gram sample of  
12 polyalphaolefin.
- 13 15. A composition according to Claim 13 wherein the composition has a  
14 Bromine Index of less than 50 mg Bromine per 100 gram sample of  
15 polyalphaolefin.
- 16 16. A composition according to Claim 13 wherein the composition has a  
17 Bromine Index of less than 25 mg Bromine per 100 gram sample of  
18 polyalphaolefin.
- 19 17. The composition of Claim 13 wherein the composition is an engine oil  
20 lubricant.
- 21 18. The composition of Claim 13 wherein the composition is a gear  
22 lubricant.

- 1 19. The composition of Claim 13 wherein the composition is an hydraulic  
2 lubricant.
- 3 20. The composition of Claim 13 wherein the composition is a compressor  
4 lubricant.
- 5 21. The composition of Claim 13 wherein the composition is an aerospace  
6 jet lubricant.
- 7 22. The composition of Claim 13 wherein the composition is a fiber optic  
8 cable gel.
- 9 23. The composition of Claim 13 wherein the composition is a synthetic  
10 grease.
- 11 24. The composition of Claim 13 wherein the composition is a dielectric  
12 fluid.
- 13 25. A method of producing a highly oxidatively stable polyalphaolefin  
14 comprising the step of hydrogenating polyalphaolefin to a level of  
15 hydrogenation in which an RBOT level of at least 2200 minutes is  
16 achieved when diphenyl amine is used as an antioxidant.
- 17 26. A method of producing a highly oxidatively stable polyalphaolefin  
18 comprising the step of hydrogenating a polyalphaolefin to a level of  
19 hydrogenation in which a Lube Oil Oxidator level of at least 45 hours is  
20 achieved when pressures between 350 and 2500 psi are applied.
- 21 27. The method of claim 1, wherein the PAO is distilled prior to  
22 hydrogenation.

- 1 28. The method of claim 1, wherein the PAO is hydrogenated and distilled
- 2 prior to the hydrogenation to a Bromine Index of less than 200.

1

ABSTRACT OF THE DISCLOSURE

- 2 The present invention relates to a process of producing a highly oxidatively  
3 stable polyalphaolefin by hydrogenating a polyalphaolefin to greatly decrease  
4 its level of unsaturation.

FIGURE

Overlay of Oxidation Analyses from Gunset et al and Highly Saturated PAO

